

# Investigation of mural painting materials discovered in the Ikh Bulag ruin of the Tuvshinshiree, Sukhbaatar Province, Mongolia

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wall, something resembling white chalk was preserved [Tsogtbaatar et al. 2019]. A sample of this white material was also taken.

## II . Samples and analytical methods

### 2.1 Samples

The fragment samples from the ruin are shown below in Figures 2, 3, 4, and 5. To study the pigments and materials used in the mural painting fragments found at Ikh Bulagiin Öndör Dovjoo, both sides of the green and red-pigmented paints were examined along with the white material.

Three types of samples were chosen and identified as “No. 1,” derived from the green pigment (Figure 2\_a, b).



Figure 1. (c) Location of mural fragments found within the ruin

Sample “No. 2” is the crushed white material (Figure 3). In addition, the red pigment was identified as “No. 3a” (Figure 4) and its opposite section was identified as “No. 3b” (Figure 5).

### 2.2. Features of the mural painting samples

The wall painting’s structure was divided into a paint layer, plaster layer, and clay or mortar layer, which was the deepest. In some murals, mortar can be up to two layers thick, and without any plaster. The layer structure of Ikh Bulagiin Öndör Dovjoo’s sample No. 1 is shown in Figure 6. The sample had a total thickness of approximately 12.95mm and consisted of paint, plaster, and mortar layers. B1, the paint layer, contains pigments of green, black, white, and red. Beneath the plaster layer,



Figure 1. (d) Fragments of mural painting



Figure 2. Sample No. 1 (a) front; (b) back



Figure 3. Sample No. 2



Figure 4. Sample No. 3a (Front side)



Figure 5. Sample No. 3b (Back side)



there was another layer of paint with red pigment (layer B<sub>2</sub>).

The layer structure of sample No. 3 is shown in Figure 7. It has two paint layers on both sides. The total thickness of the sample is approximately 18.5mm. The P3 plaster layer in Figure 7, has a thickness of 2.05–2.9 mm and the mortar layer (M<sub>2</sub>) has a thickness of approximately 2.8mm.

### 2.3. Analytical methods

Basic tools rented from the Center for Archaeological Operations of the Nara National Research Institute for Cultural Properties of Japan were used for the investigation. These devices were as follows: optical microscopy, X-ray fluorescence analysis (XRF) for revealing elements contained in minerals in material, X-ray diffractometry (XRD) for determining mineralogical crystals; scanning electron microscopy/energy dispersive X-ray fluorescence analysis (SEM/EDX) for recognizing pigment particle sizes, and X-ray radiography for testing inner structures.

#### 2-2-1. Optical microscopy

The surface appearance and shape of the pigments were observed using stereo microscopes (Leica Z16 APOA, Leica MZ16, and OMRON Corporation VCR-FRM20LZ). The observation distance from the objective lens to the object was 30–150mm, with a magnification of 10–120X.

#### 2-2-2. X-ray fluorescence analysis (XRF)

Elements of the mural plaster and pigments were measured using two separate XRF analyzers. For sample fragments of mural paintings, a desktop-type analyzer (Bruker EAGLE III) was used. For the mural paintings in situ, a handheld, nondestructive-type, NITON XL3t was similarly employed. The measurement conditions for the desktop analyzer were molybdenum target, tube voltage, current set to 40 kV and 30  $\mu$ A, and a 100 s acquisition time. The conditions for the NITON XL3t were gold target, tube voltage 40 kV, tube current controlled automatically, and a 100 second acquisition time.

#### 2-2-3. X-ray diffractometry

The mural plaster and pigments were identified by

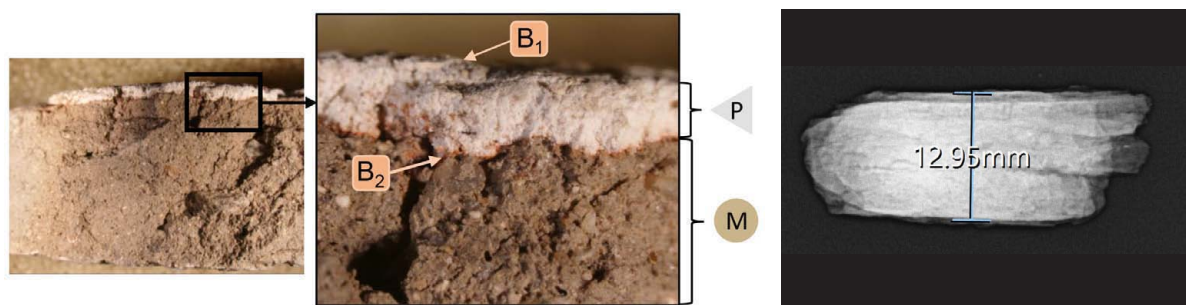


Figure 6. Layer structure of sample No. 1.

Right side monochrome photograph is an X-ray radiograph. Letter mark: paint layer (B<sub>1</sub>, <sub>2</sub>); plaster layer (P); mortar layer (M)

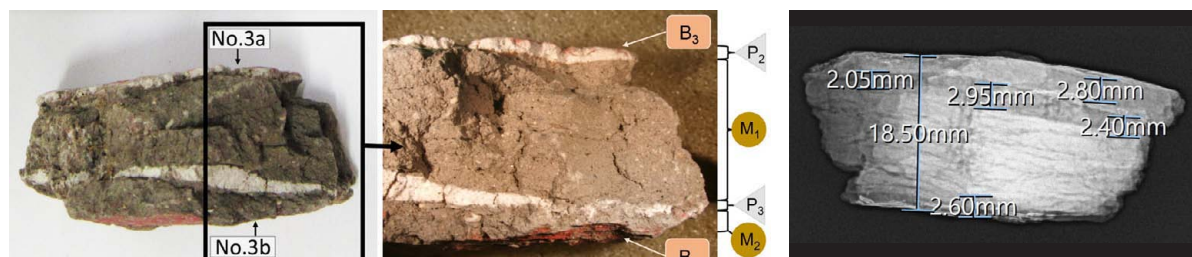


Figure 7. Layer structure of sample No. 3.

Right side monochrome photograph is an X-ray radiograph. Letter mark: paint layer (B<sub>3</sub>, B<sub>4</sub>), plaster layer (P<sub>2</sub> and P<sub>3</sub>), mortar layer (M<sub>1</sub> and M<sub>2</sub>)

XRD, Rigake Smartlab. The measurement conditions were as follows: Cu-K alpha radiation, tube voltage 40 kV, tube current 40 mA, and scan range ( $2\theta/\theta$ ) 5–90°.

#### 2-2-4. Scanning electron microscopy

The shape and elements of the pigment particles were observed and analyzed using SEM/EDX, JEOL JSM-IT100. The observation magnification was x30–x2000.

### III . Results and discussion

#### 3.1. Sample No. 1

The green pigment in layer B<sub>1</sub> of sample No.1 excavated from Ikh Bulagiin Öndör Dovjoo, contained high amounts of the elements Cu and Cl in the XRF. The green pigment was identified as “atacamite”  $\text{Cu}_7\text{Cl}_4(\text{OH})_{10}\cdot\text{H}_2\text{O}$  by XRD.

Results of the XRD are shown in Figure 8. Atacamite is known to have been used as a pigment in Asia and the West [FitzHugh et al. 2003] In some additions, atacamite has been identified from green pigment in Mongolia and Inner Mongolia [Ryu 2018; Wei et al. 2010; Du et al. 2002].

It has been shown that pigments may be identified from the type of material particles. Crystalline structures

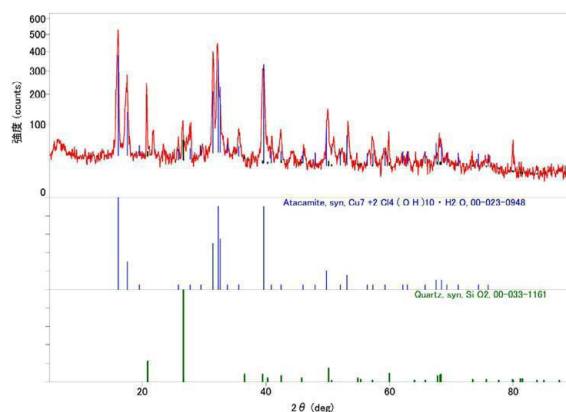


Figure 8. Results of the XRD analysis on green pigment from sample No. 1

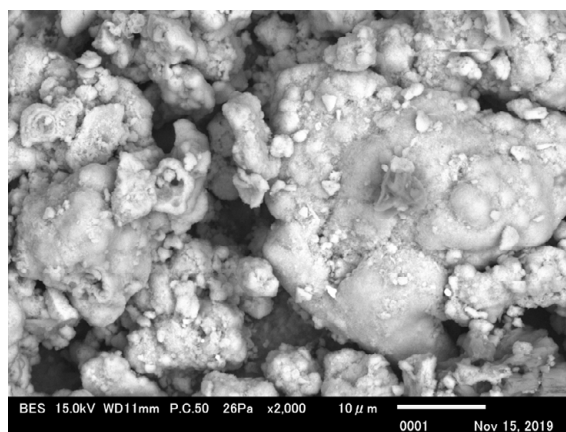


Figure 9. Scanning electron micrograph of green pigment from sample No. 1, magnification 2000x

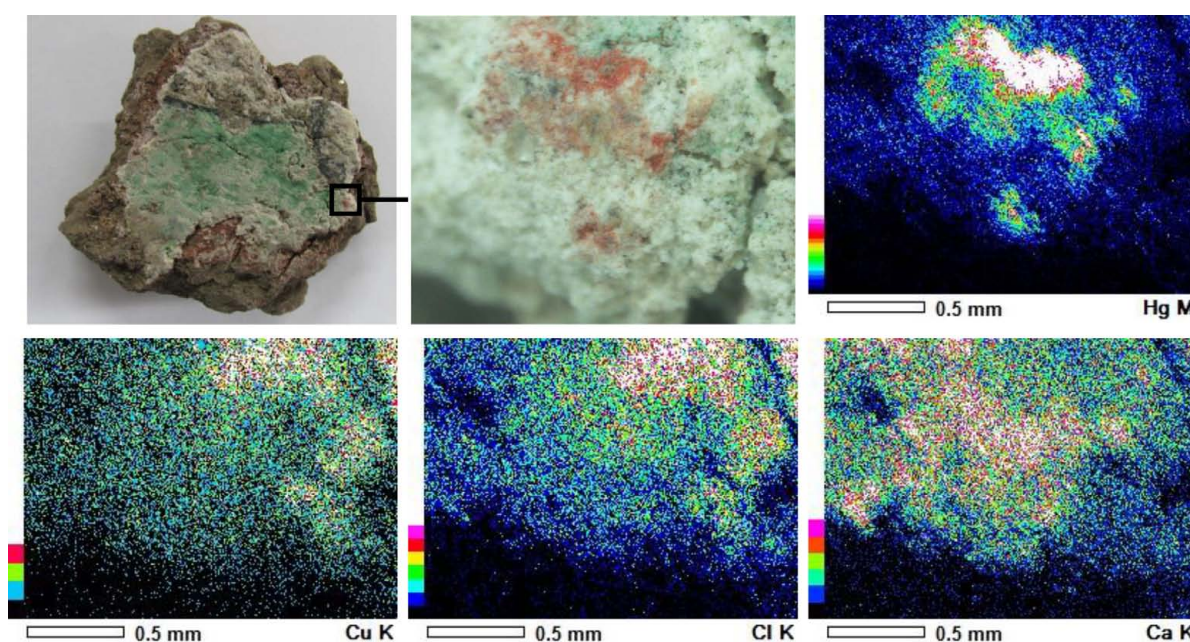


Figure 10. The results of SEM/EDX element mapping from sample No. 1, red pigment. Measurement point: No. 1\_05 of layer B<sub>1</sub>



determine the shape of pigment particles. The green paint in layer B<sub>1</sub> was observed at magnifications from 30-2000X on a scanning electron microscope (SEM), and the pigment particle was determined. The SEM image showed edgeless and smooth particles, common in paint with copper chloride hydroxide, “atacamite.” The particle shape and sizes were similar to the Övgön temple in Bulgan, Mongolia [Ryu 2020]. The particle size was approximately 1–5 μm. Results of the SEM are in Figure 9.

Table 4 in Appendix 3 shows the results from XRF analysis of samples No. 1, No. 3a, and No. 3b. The elements Ca, Pb, As, Si, Fe, and Cu were detected from the white pigment at point No. 1\_02 of layer B<sub>1</sub> of the fragment. The high level of Ca detected may be related to the material components of the plaster beneath the paint layer. In addition, because Pb was detected from point No. 1\_02, white lead was estimated to be the pigment. Hg was revealed under both X-ray fluorescence analysis and SEM/EDX element mapping (Figure 10) of the red pigment at point No.1\_05 of layer B<sub>1</sub>. According to X-ray diffractometer analysis, it is definitive that cinnabar HgS red paint was used.

High Fe content was detected in the red pigment in layer B<sub>2</sub> at points No. 1\_06 and No. 1\_07. It is likely hematite Fe<sub>2</sub>O<sub>3</sub>. “Calcite,” CaCO<sub>3</sub> was detected from the plaster layer of sample No. 1. Measurement points for XRF and XRD are shown in Appendix 2: Figure 18, 19 and 20, as well as the results of these analyses are shown in Appendix 3: Table 4 and Table 5.

### 3.2. Sample No. 2

Orthoclase Al Si<sub>3</sub>O<sub>8</sub> and quartz SiO<sub>2</sub> were detected from sample No. 2 using an X-ray diffractometer. The results of X-ray diffractometer analysis are shown in Figure 11.

### 3.3. Sample No. 3

After preparing the cross-sectional sample No.3, we examined the paint layer and the underlying structure. As a result of the optical microscopy, it was determined that section No.3a was a paint layer (B<sub>3</sub>) on the top, and the second layer was a plaster layer (P<sub>2</sub>). The third layer has

been identified as a layer of red paint (B<sub>5</sub>).

In addition, section No.3b consisted of three different layers: a paint layer, a mortar layer, and a plaster layer. The first layer was a paint layer (B<sub>4</sub>) on section No.3b, the mortar layer (M<sub>2</sub>) was under the B<sub>4</sub> layer, and the third layer was a plaster layer (P<sub>3</sub>). Figure 12 shows the cross-sectional image of sample No.3 and its layer structures.

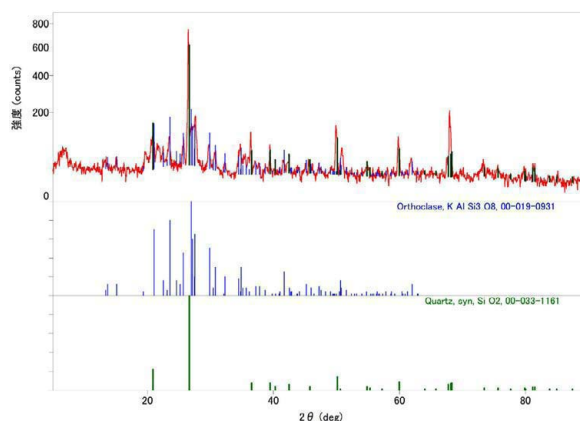


Figure 11. The results of XRD analysis on sample No. 2

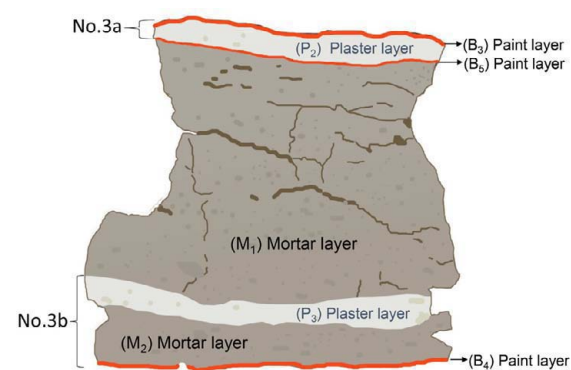


Figure 12. A cross-section image of sample and illustration of its layer structures

XRF, XRD analysis, and SEM/EDX were performed on many of the layers of sample No.3. As a result, a high amount of Si was detected from the plaster layer ( $P_2$ ,  $P_3$ ) at points No. 3a\_02, 03, and 3b\_7 by XRF analysis. The XRD analysis revealed that the plaster layers at point No.3a\_11 and No.3b\_13 were Orthoclase  $Al\ Si_3O_8$  (Appendix 3, Table 5).

High Hg content was detected by XRF analysis and SEM/EDX mapping of the red pigment on layer B3 of sample No. 3a (Figure 15) and layer B4 of sample No. 3b. The XRF results are shown in Figures 13 and 14. The red pigment from B3 and B<sub>4</sub> was identified as cinnabar<sup>(1)</sup>(vermilion<sup>(2)</sup>)  $HgS$  by XRD analysis. Cinnabar is a well-known pigment in the art and cultural heritage fields. There are three kinds of mercuric sulfide pigments, such as the natural mineral form, which is simply finely ground cinnabar and a synthetic form, which is produced by the dry process, as well as a synthetic form, which is produced by a wet process. The dry-process vermilion was in common use, and was well

- (1 The mineralogist and crystallographer have given the common red crystalline form of mercuric sulfide the name cinnabar [Gettens et al. 2012:159-182].  
(2 Vermilion is the standard name in England [Gettens et al. 2012: 159-182].

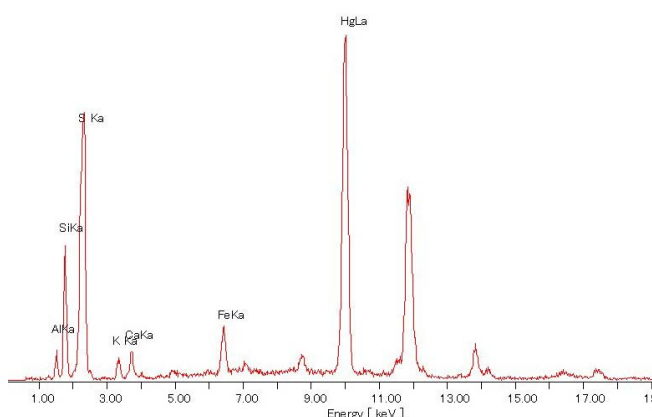


Figure 13. The results of x-ray fluorescence analysis on sample No. 3a.

Measurement point: sample No. 3a\_01

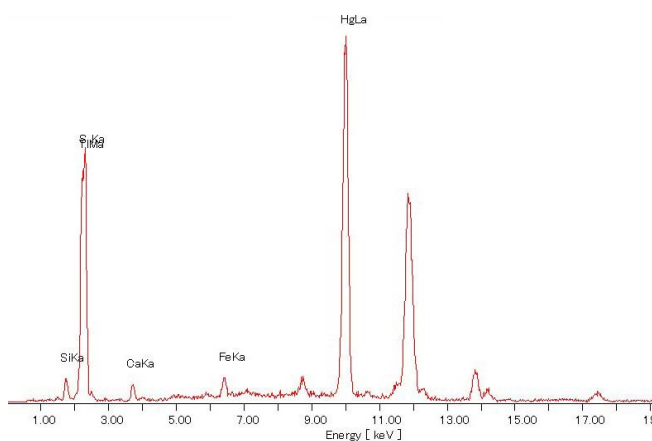


Figure 14. The results of x-ray fluorescence analysis on sample No. 3b.

Measurement point: sample No. 3b\_04

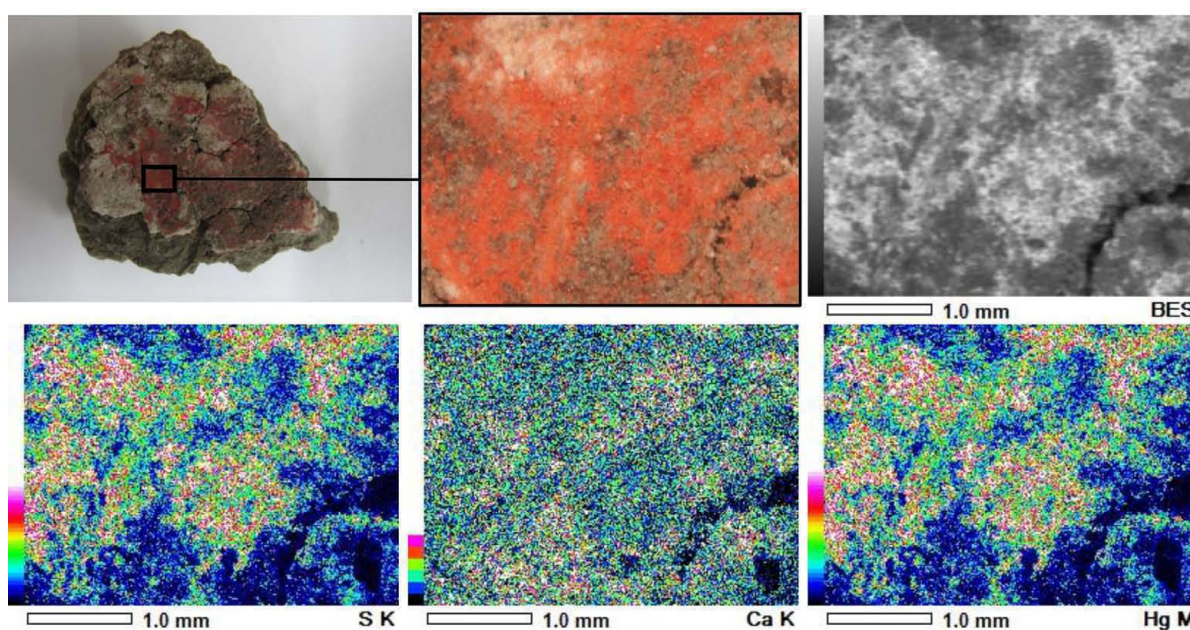


Figure 15. The results of element mapping with SEM/EDX on sample No. 3a, red pigment.

Measurement point: red pigment from layer B<sub>3</sub>



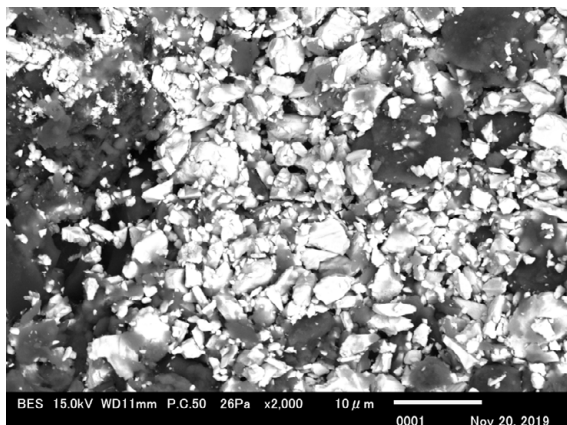


Figure 16. Scanning electron micrographs of red pigment from sample No. 3a, magnification 2000x

developed in China by the 17th century. Gradually, knowledge of the production and manufacture of the pigment may have been transmitted and imported to Western and Asian countries from China [van Vallen 2005].

This type of red paint is used for some murals, including the Kharkhorum ruin [Angaragsuren 2020], Övgön Monastery [Ryu 2020], Tsamba shrine of the Erdene Zuu monastery in Mongolia [Angaragsuren 2018], and Dazhao temple in Inner Mongolia, China [Wei et al. 2010; Du et al. 2002]. It is clear that the particle sizes of the red pigment used in the mural painting of the Ikh Bulagiin Öndör Dovjoo ruin were similar to the particle size of the red pigment in the mural painting of the Kharkhorum ruin and Övgön monastery. In addition, it was estimated that the red pigment was produced by a dry method when examining the shape of the pigment particles using SEM.

The particle sizes of red pigment in both section No. 3a and 3b were prismatic and approximately 1–10 μm in size. SEM examination results were compared between sections 3a and 3b, and pigment particle sizes of section No. 3b were larger than the pigment particle size of section No. 3a. Micrography of the pigment particles is shown in Figures 16 and 17.

#### IV . Conclusion

In conclusion, the materials and pigments used in mural paintings excavated from the Ikh Bulagiin Öndör Dovjoo ruin of Sukhbaatar Province, Mongolia, were

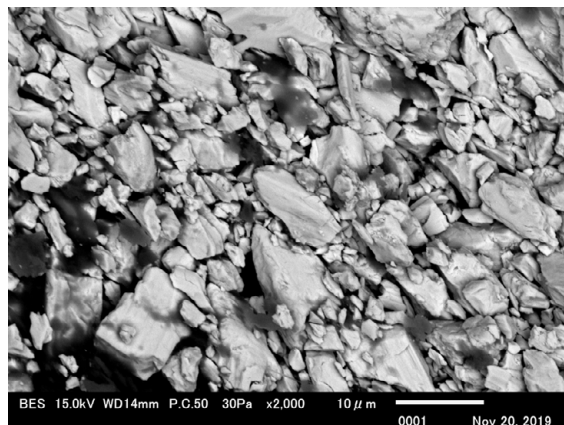


Figure 17. Scanning electron micrographs of red pigment from sample No. 3b, magnification 2000x

successfully investigated. Two primary conclusions can be reached:

##### 1) Investigation of the materials and pigments

The green paint in fragments of the mural painting was atacamite ( $\text{Cu}_7\text{Cl}_4(\text{OH})_{10}\cdot\text{H}_2\text{O}$ ), the plaster layer was calcite ( $\text{CaCO}_3$ ), and the red paint was cinnabar ( $\text{HgS}$ ). The element Pb was detected within the white paint layer at point No. 1\_02 and was estimated to be white lead pigment.

##### 2) For further research and excavation

Visual observation of sample No. 1 reveals that it was part of a multi-colored mural. Therefore, when re-excavating this ruin, the original mural pieces should be removed without damage. It may be possible to reassemble the small fragments into a whole.

It is apparent that sample No. 3 has paint layers on both sides with plaster in between. It can be assumed that the surface of the mural painting peeled off and fell forward causing overlap. This should be kept in mind during future excavations.

The results of this investigation can be used to aid future researchers when comparing ancient historical sites and ruins.

It can be observed that the materials used for wall paintings at Sumiin Dovjoo and Ikh Bulagiin Öndör Dovjoo, which are geographically quite close, were very different. This finding can assist other researchers investigating pigment materials and conserving cultural heritage sites.



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## Abbreviation:

MAS: Mongolian Academy of Sciences

АХГБСХ: Археологийн хүрээлэнгийн гар бичмэлийн сан хөмрөг (Archive document of the Institute of Archaeology)

УБ: Улаанбаатар (Ulaanbaatar)

ШУА: Шинжлэх Ухааны Академи

# ■ APPENDIX 1:

Results of the optical microscopy of sample No. 1, No. 3a, and No. 3b

Table 1. Results of optical microscopy on sample No. 1

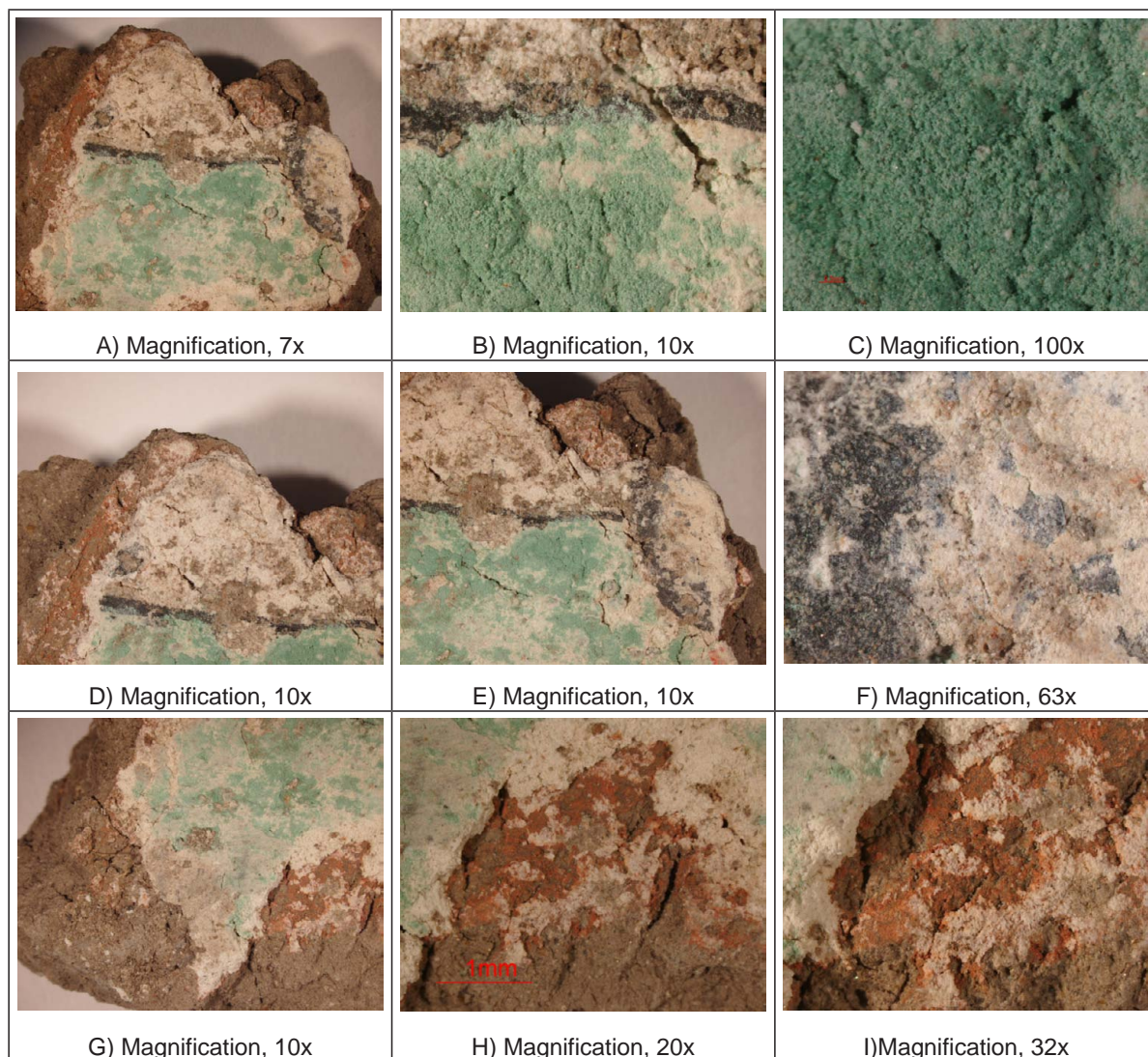


Table 2. Results of optical microscopy on sample No. 3a

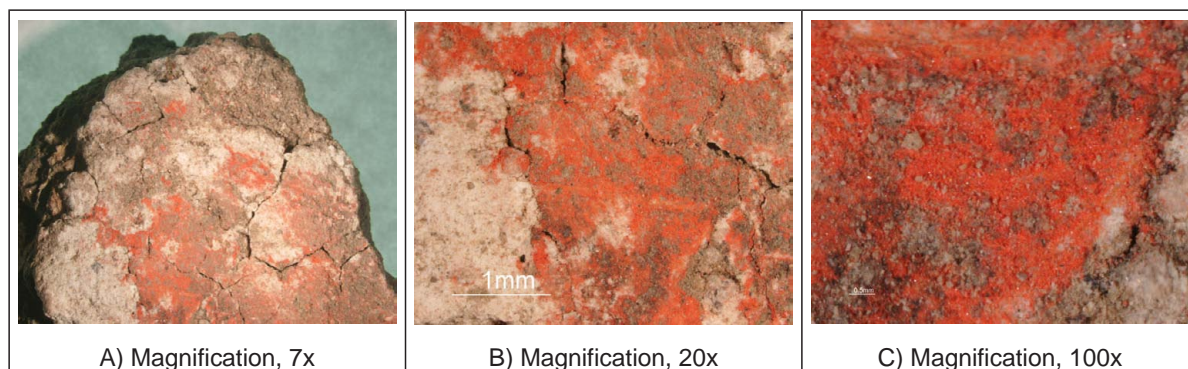
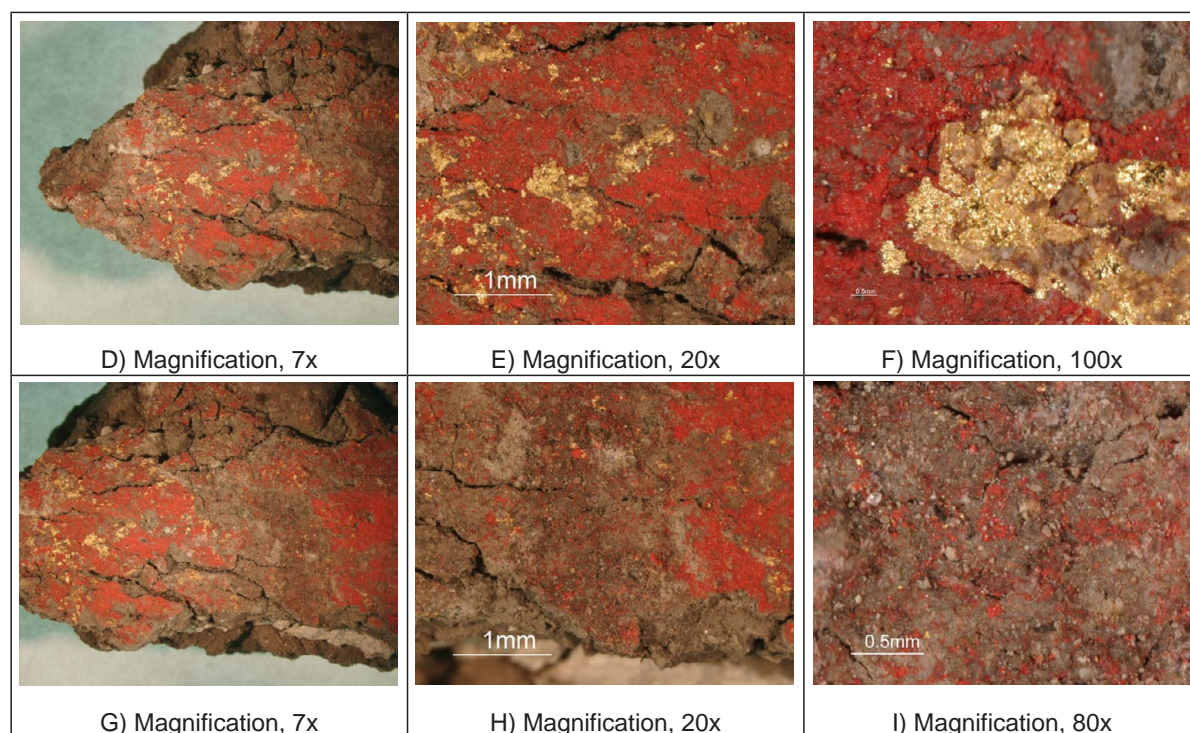




Table 3. Results of optical microscopy on sample No. 3b



## ■ APPENDIX 2:

XRF and XRD measurement points on samples No. 1, No. 3a, and No. 3b

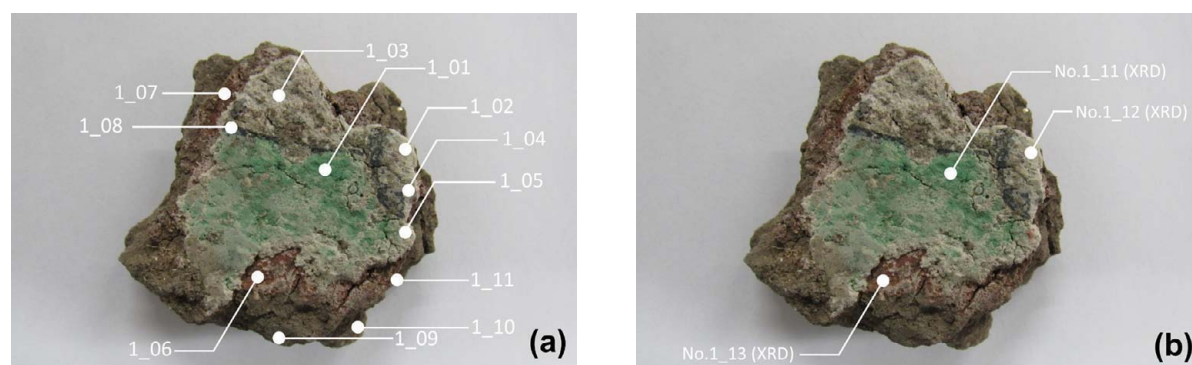


Figure 18. (a) XRF measurement points on sample No. 1; (b) XRD measurement points on the sample No. 1

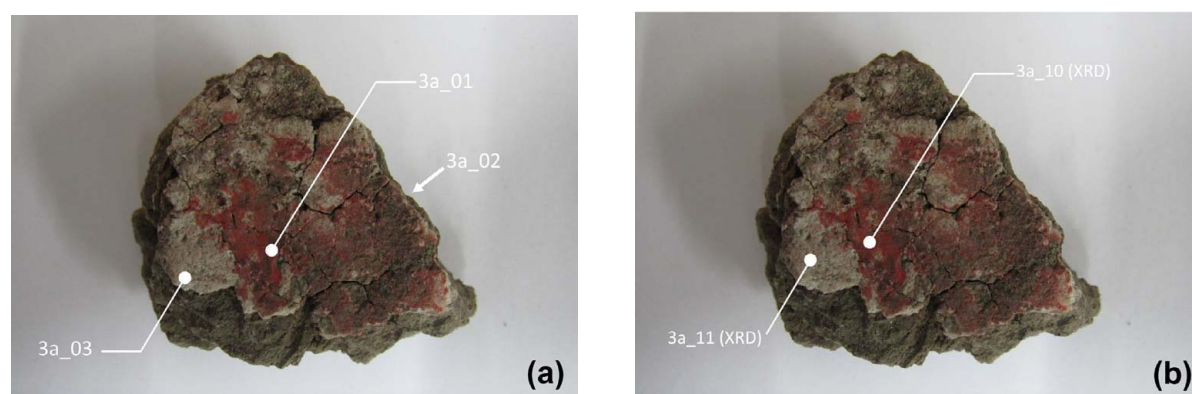


Figure 19. (a) XRF measurement points on sample No. 3a; (b) XRD measurement points on the sample No. 3a



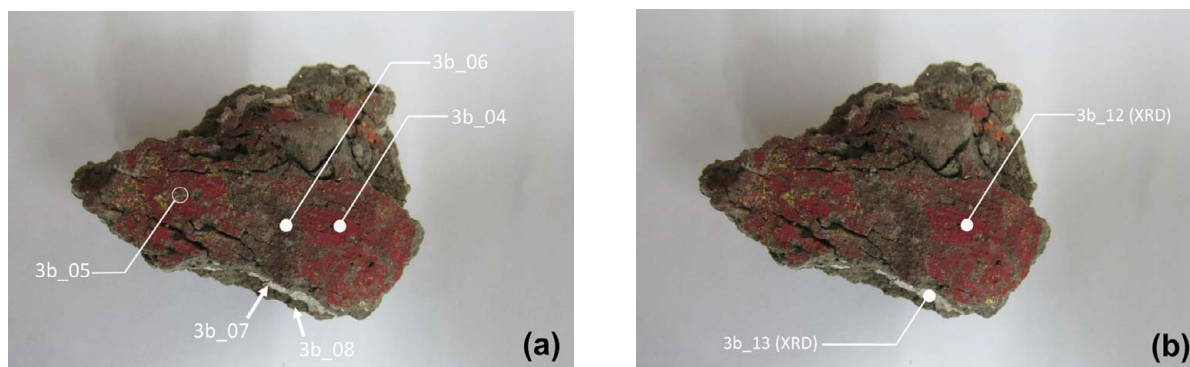


Figure 20. (a) XRF measurement points on sample No. 3b; (b) XRD measurement points on sample No. 3b

### ■ APPENDIX 3:

Results of XRF and XRD analysis of the Ikh Bulagiin Undur Dovjoo mural painting

Table 4. Results of x-ray fluorescence analysis on sample No. 1, No. 3a, and No. 3b

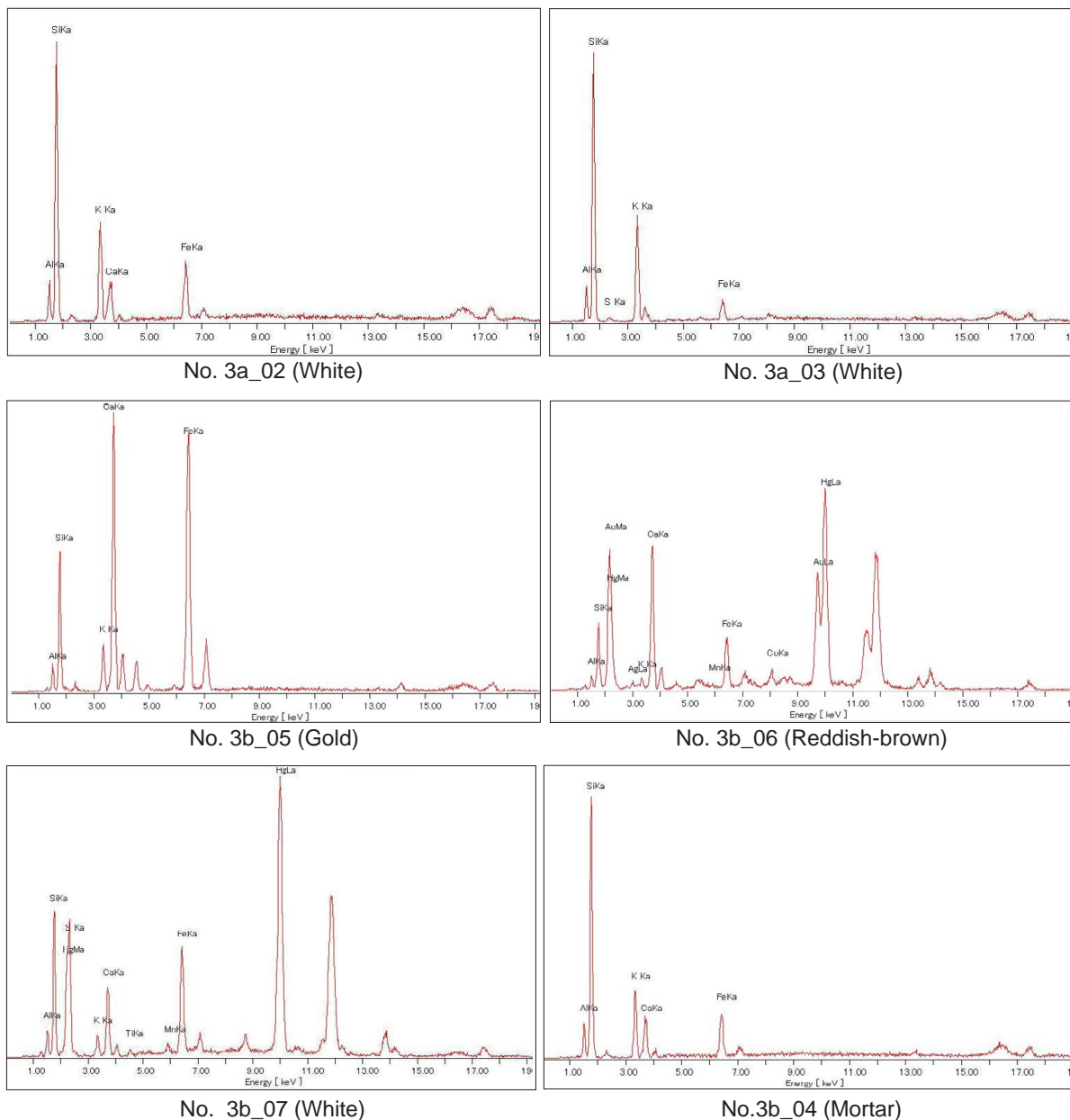
Sample No.	Measurement point	Detected elements
No. 1_01	Green	Cu, Cl, S, Fe, Ca
No. 1_02	White	Ca, Pb, As, Si, Fe, Cu
No. 1_03	White	Si, K, Fe, Cu, Al, Ca, Cl, S, Mn
No. 1_04	White	Si, K, Fe, Al, Ca, Ti, Cu, Pb
No. 1_05	Red	Si, S, Hg, K, Fe, Al, Cu
No. 1_06	Red	Ca, Si, Fe, Al, K, Cr, Cu, Ti, S, Mg
No. 1_07	Red	Fe, Ca, Si, K, Al, Ti, Cu
No. 1_08	Black	Si, K, Fe, Cu, Ca, Al, S, Cl, Ti
No. 1_09	Mortar	Ca, Fe, Si, Cu, K, Ti, Al, S, Mn
No. 1_10	Mortar	Ca, Fe, Si, K, Al, Ti
No. 1_11	Mortar	Ca, Fe, Si, Al, K, Ti, Mn
No. 3a_01	Red	Hg, S, Si, Fe, Al, Ca, K
No. 3a_02	White	Si, K, Fe, Ca, Al
No. 3a_03	White	Si, K, Al, Fe, S
No. 3b_04	Red	Hg, S, Ti, Si, Ca, Fe
No. 3b_05	Gold	Hg, Ca, Au, Si, Fe, Cu, Al, K, Ag, Mn
No. 3b_06	Reddish-brown	Hg, Si, S, Fe, Ca, Al, K, Mn, Ti
No. 3b_07	White	Si, K, Fe, Ca, Al
No. 3b_08	Mortar	Ca, Fe, Si, K, Al

Table 5. Results of XRD analysis on sample No. 1, No. 3a, and No. 3b

Sample No.	Measurement point	Detected elements
No. 1_11 (XRD)	Green	Atacamite $\text{Cu}_2\text{Cl}_4(\text{OH})_{10} \cdot \text{H}_2\text{O}$ , quartz $\text{SiO}_2$
No. 1_12 (XRD)	White	Aragonite $\text{Ca}(\text{CO}_3)$ , Calcite $\text{CaCO}_3$ , quartz $\text{SiO}_2$
No. 1_13 (XRD)	Red	Quartz $\text{SiO}_2$ , Hematite ? $\text{Fe}_2\text{O}_3$
No. 3a_10 (XRD)	Red	Cinnabar $\text{HgS}$ , quartz $\text{SiO}_2$
No. 3a_11 (XRD)	White	Orthoclase $\text{K Al Si}_3\text{O}_8$ , quartz $\text{SiO}_2$
No. 3b_12 (XRD)	Red	Cinnabar $\text{HgS}$
No. 3b_13 (XRD)	Plaster layer	Orthoclase $\text{K Al Si}_3\text{O}_8$ , quartz $\text{SiO}_2$

## ■ APPENDIX 5:

Results of x-ray fluorescence analysis on sample No. 3a and No. 3 (Graphic data)



### Abstract:

Монгол улсын Сүхбаатар аймгийн  
Түвшинширээ сумын Их Булагийн тууриас  
илэрсэн ханын зургийн материалын  
шинжилгээ

Одхүүгийн Ангарагсүрэн  
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малтан судалжээ. Малтлагаас илэрсэн ханын  
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(SEM/EDX) багажийг тус тус ашиглалаа. Судалгааны үр дүнд No.1 дээжийн ногоон хэсгээс атакамайт ( $\text{Cu}_7\text{2Cl}_4(\text{OH})10\cdot\text{H}_2\text{O}$ ), No.3 дээжийн улаан хэсгээс мөнгөн усны хүдрийн будаг ( $\text{HgS}$ ) тус тус илэрч, суурь давхарга нь кальцит ( $\text{CaCO}_3$ ) болох нь тогтоогдов.

モンゴル国スフバートル県トウブシンシレー郡  
に所在するイフ・ボラギーン・ウンドウル・ド  
ブジョー遺跡で発見された壁画材料の分析

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モンゴル科学アカデミー歴史学・考古学研究所と大阪大学は「東部モンゴルの突厥期の歴史・考古学研究」プロジェクトに基づき、スフバートル県トウブシンシレー郡にあるイフ・ボラク川の石築基遺跡（ドブジョー (Dovjoo)）を発掘した。本研究は発掘中に発見された壁画サンプルに対して顔料と材料の分析を行ったものである。分析は光学顕微鏡、蛍光X線分析 (XRF)、X線回折分析 (XRD)、走査型電子顕微鏡観察 (SEM) の手法を用いて壁画材料の同定を行なった。分析の結果、サンプル No.1 の緑色部分からはアタカマイト ( $\text{Cu}_7\text{2Cl}_4(\text{OH})10\cdot\text{H}_2\text{O}$ ) が、サンプル No.3 の赤色部分からは水銀朱 ( $\text{HgS}$ )、漆喰層からはカルサイト ( $\text{CaCO}_3$ ) が検出された。

(67 頁よりつづく)

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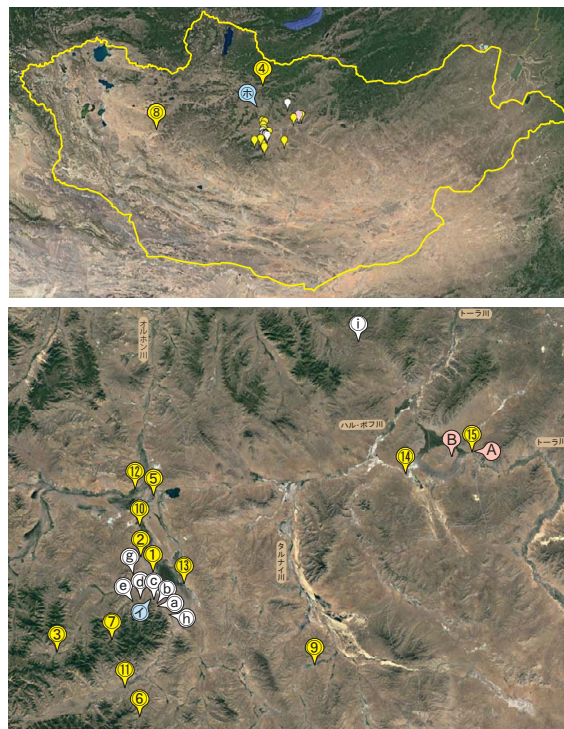
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### 5. 言及されている遺跡

A: ザーマル古墳 (僕固乙突墓) B: オラーン・ヘレム壁画墓 (①～⑮, ①, ②, ③は目次記載遺跡に対応。④は③、⑤は④、⑥は④付近か範囲内。⑦は不明)

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なお、図 1,2 報告書の書誌情報は村上論文参照。(大谷)